Standard Operating Procedure for Analysis of Nitrogen in Fertilizer Using the Leco FP428 Combustion Analyzer

1.0 Scope and Application

This is a **recommended** procedure to follow for using the Leco FP-428 for the combustion of nitrogen in fertilizers. The procedure that follows is for finding nitrogen in fertilizer. This can also be modified for finding protein in feeds. This method is applicable for measuring nitrogen in the range of 1-67%.

2.0 Summary of Method

The instrument is calibrated with a standard reference material which happens to be uric acid. About 0.1000 g of sample is placed into a tinfoil cup and then 0.2000 g of sucrose is added. Use a check sample every tenth sample to make sure there is no analytical drift. Always run a few blanks to make sure it is at the correct calibration and to recalibrate if necessary whenever the sealed combustion tube is exposed to air.

3.0 Definitions

The definitions and purposes below are specific to this method, but have been conformed to common usage as much as possible.

3.1 Grams-g

3.2 LIMS-laboratory information management system

3.3 May: This action, activity, or procedural step is neither required nor prohibited

May not: This action, activity, or procedural step is prohibited

Must: This action, activity, or procedural step is required

Shall: This action, activity, or procedural step is required

Should: This action, activity, or procedural step is suggested, but not required

4.0 Interferences

No interferences are known at this time.

5.0 Safety

5.1 The toxicity or carcinogenicity of each analyte or reagent has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.

5.2 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 Equipment and supplies

6.1 Note: Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.

6.2 Analytical balance. Anything that reads 4 places beyond the decimal point.

6.3 Tin foil cups.

6.4 Tin capsules.

6.5 Forceps.

6.6 A combustion analyzer.

7.0 Reagents and Standards

7.1 Sucrose. 99%+ pure.

7.2 Uric acid. 99%+ pure.

7.3 Ammonium Dihydrogen Phosphate

8.0 Sample Collection, Preservation, and Storage

8.1 Collection is done by the Department of Agriculture. The inspectors collect a

small sample that is a good representation of the whole lot. The samples are then brought down by the Department of Agriculture. Samples are logged into the LIMS at the lab and are stored on the shelves in the grinding room after being riffled by the chemist in charge. The samples are then kept there until they have either passed or met their holding times after they have been declared deficient.

9.0 Quality Control

9.1 The laboratory participates in the Magruder fertilizer and Association of Florida Phosphate Chemists check sample programs. Both of these check sample programs distribute check samples monthly and results are compared to up to 81 other labs participating in the analyses.

9.2 The control sample is ammonium dihydrogen phosphate. Weigh about 0.1000 g of the control sample into a tinfoil cup and then add about 0.2000g of sucrose. Four control samples should be run immediately after calibration to make sure the calibration is correct. The sample should read 12.15% +/- 0.2%. Then the control should be used after a set of ten regular samples.

9.3 The combustion analyzer needs to be set up for running air blanks. A minimum of 15 need to be run. The analyzer must be reading 0.000 or as close to it as possible. This means that an adjustment may be needed on the manual blank until it reads 0.000 or close to it.

9.4 A duplicate and a check is analyzed with each group of ten samples.

10.0 Calibration and Standardization

10.1 The combustion analyzer should be set to the specifications that are the manufacturer's recommended operation conditions. There is a section in the instrument operation manual that talks about fertilizer samples, and these are the conditions that should be used.

10.2 To begin calibration, the blank must be as close to 0.000 as the controller wants while making sure the control sample is within its limits. Then four samples of uric acid are run through. Each one weighing about 0.1000 g with NO sucrose added. Then choose those four results to calibrate the instrument. The uric acid should be 33.327. Then run four check samples (ammonium dihydrogen phosphate) to make sure it is running in range. The range for this particular check is 11.95% to 12.35%. If it is not, repeat the procedure again.

10.3 The system used for calibration is stored in the analyzer's own database.

Calibration should be done each time the instrument is used. The calibration is always stored from the previous run so it must be changed each time a new one is made.

11.0 Procedure

11.1 The transfer of data between the LIMS and the Leco can be a tricky procedure. This will be a step by step account of how to transfer the data and also how to run the combustion analyzer.

11.2 Go to the Toleco icon and create a work list using analyte #44601.

11.3 Using the already ground samples, weigh each one into a tinfoil cup to the eight of about 0.1000 g and then add about 0.2000 g of sucrose. Use a duplicate and a check every ten samples.

11.4 After obtaining the weights of the sample that are weighed, call up the work list fill it and then distribute.

11.5 DO NOT COMPLETE DISTRIBUTION YET! Wait until the data has been transferred to the Leco with no problems.

11.6 Quit this and then go to instrument. At this time Export then quit and leave the LIMS.

11.7 Leaving LIMS lets the data be transferred to the disk in drive a.

11.8 Take the disk and put into the Leco. Press the login icon.

11.9 Go to weights and insert file icons.

11.10 Then push load and overwrite the old file on the Leco.

11.11 After the file has loaded, go to ID's find HALT and hit OUT OF SEQUENCE 3 or 4 times.

11.12 Go back to ID's, find Blank and hit OUT OF SEQUENCE at least 10 times.

11.13 Wait for the results to hit zero or just about zero. Try to keep it at 0.001 from 0.000 with at least one blank.

11.14 Do not put the carousel on while running the blanks.

11.15 Run the first four uric acid samples. During the last acid sample run, push the switch from auto to manual. Now standardize.

11.16 Go to VIEW, SYSTEM FOLDERS, SYSTEM UPDATE, and to the standard calibration icon.

11.17 Select the four uric acid samples that have just been run and then push the PROCESS RESULTS icon then print. Escape back to the analyze screen.

11.18 Go to SYSTEM FOLDERS, SYSTEM OUTPUT, DATABASE TRANSFER, and then include all the samples that are needed for transfer back to the LIMS.

11.19 Go ahead and overwrite the file. Just follow the steps until everything is processed back onto the disk. Put the disk back into the computer disk drive.

11.20 Use the FromLeco icon.

11.21 Call up the work list for the samples that were just run. Distribute and then complete distribution.

11.22 Go to Instrument, Import, New. Then make the analyte #22102.

11.23 Look at the samples and make sure everything is correct. Distribute the work list and then complete distribution.

11.24 Sign off on the bench sheet and put it in the nitrogen binder.

12.0 Data Analysis, Calculations, and Reporting Results

12.1 Calculations: All calculations are done by the computer based on the calibration.

12.2 Reporting Results: All results reported are in a weight/% format. The LIMS decides if the sample is deficient or not. If it is, it is run in duplicate the next time.

13.0 Method Performance

No method performance data is available for this method.

14.0 Pollution Prevention

No information is available at this time.

15.0 Waste Management

15.1 Giving samples away after they have spent the necessary time of holding limits the amount that is thrown away.

15.2 For further information on waste management consult The Waste Management Manual for Laboratory Personnel and Less is Better: Laboratory Chemical Management for Waste Reduction, both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th street N.W., Washington, D.C. 20036

16.0 References

Official Methods of Analysis (1995) 16th Ed., AOAC, Washington, D.C.Section 2.4.02